

## **Statement of Work for Organics Analysis Comments and Responses Summary Document**

This document is a summary of comments received by USEPA regarding the Contract Laboratory Program (CLP) Statement of Work for Organics Analysis, SOM01.X (5/2004). Comments were received at or subsequent to the *Superfund Analytical Operations Training Conference* in June 2004. USEPA's responses to each of the comments are contained in this document. When appropriate, changes have been incorporated into the Statement of Work for Organics Analysis, SOM01.0 (8/2004). Please note that comments that refer to multiple fractions are located within the General Comments section of this document.

### **General Comments**

1. **Statement: When will the CCS checker be made available for the new SOW?**

The complete CCS checker will be available at the end of Calendar Year (CY) 2004 to all laboratories and software vendors. At this point it is unclear if a beta version will be operational at the time of pre-award.

2. **Statement: How will price submissions be evaluated? Is there a formula, and will it be made available? If so, when?**

This question is related to the Request for Proposal (RFP) and will be addressed by USEPA OAM.

3. **Statement: A rather grandiose statement appears as 8.2.2 in several sections: "The samples must be stored in an atmosphere demonstrated to be free of all potential contaminants.". How? How often?**

No additional language is necessary as this is to serve as a guideline. This is a general guideline and as long as storage blanks do not show any contamination, then the atmosphere has been shown to be sufficiently free of all potential contaminants.

4. **Statement: VOAs, Container/volume question for field samplers guide vs. SOW (two bottles vs. four bottles).**

The Statement of Work (SOW) has been modified to indicate that volatile water samples will require only two bottles. Four vials are required for trace level volatiles that require Selected Ion Monitoring (SIM) analysis.

5. **Statement: Storage blanks for VOAs, general only for other fractions? Appropriate storage: samples away from standards, water away from soils?**

There is no requirement to store water samples separate from sodium bisulfate preserved soil samples. Non-chemically preserved samples will be stored in a freezer, which would not be appropriate for a water sample.

SOM01.X Comments and Responses

*General Comments (Con't)*

6. **Statement: I know for a fact that I cannot meet a minimum response factor of 0.01 for 1,4-dioxane using the internal standards they have listed.**

The minimum response factor has been changed to 0.005 for 1,4-dioxane for both trace and low/medium volatiles.

7. **Statement: Regarding 1,4-dioxane, the method calls for 14 deuterated surrogate compounds, making the cost of the stock standards for this analysis rather considerable.**

The cost of standards should be figured into the laboratory's potential bid price. Commercial vendors are trying to make standard mixtures that are as cost effective as possible.

8. **Statement: Has any vendor (LIMS or otherwise) produced an automated Stage 3 SEDD?**

At this time USEPA has been piloting the Staged Electronic Data Deliverable (SEDD) Stage 2B and has received acceptable electronic deliverables. USEPA is looking to pilot Stage 3 with interested vendors.

9. **Statement: Are any commercially available software packages available to produce a SEDD that conforms to Section H and/or the form set defined in section B of the latest Draft SOM01.X?**

The SEDD Tool is free. Most database software can create Extensible Markup Language (XML) files to use for SEDD.

10. **Statement: If so, are the only commercially available products bundled in a LIMS? Does the laboratory have to purchase a LIMS to do the data reporting and EDD?**

At least one CLP vendor is producing products bundled in a Laboratory Information Management System (LIMS). Although most laboratories use a LIMS, it is not necessarily required for data reporting and the EDD.

11. **Statement: The requirement that "PDF files over 100MB should be broken down into smaller files" (B-2.8.1) is not clear. Is this a suggestion or a requirement? Most data reports will not be larger than 100MB, and those that are will be certainly be under 200MB. In our opinion, it would be easier to keep the data report in one file. The largest SDG that we have done at CEIMIC was 3800 pages and the file size was only about 125MB.**

This language has been removed. The laboratory can provide the hardcopy data in Portable Document Format (PDF) on one CD.

12. **Statement: Will USEPA consider a contract simply for PCBs?**

Chlorinated Biphenyl (CB) congeners has been removed from SOM01.X. USEPA intends to integrate CB congeners into a Dioxin analytical method.

13. **Statement: GC/MS of Aroclor is no longer required? What is the rationale? Second column mismatch for Pesticides noted but not resolved. (Has been a quality problem in the past.)**

SW-846 Aroclor identification has historically been performed by pattern recognition using Gas Chromatography (GC). Interpretation of Gas Chromatograph/Mass Spectrometer (GC/MS) results can be very confusing and possibly misleading, and it is seen as a very labor intensive process to interpret the results. If the laboratories detect samples that have high enough concentration to allow for GC/MS, they must contact the Sample Management Office (SMO) to determine if the Region wants the GC/MS analysis performed. The laboratories can contact SMO in advance if they wish to determine if the Region wants GC/MS confirmation for future samples.

The second column mismatch for pesticides has been noted but currently no resolution to this problem has been made.

14. **Statement: The new SOW has a much larger list of analyte requirements for QC compounds. Have suppliers been contacted to provide spiking cocktail solutions? If not, this will be costly for the laboratories.**

Vendors for the Deuterated Monitoring Compounds (DMCs) are aware of the new requirements of the SOW and are preparing specialized cocktails.

15. **Statement: The SOW references the use of SIM analysis. There is mention of special project requirements for this, however, it is unclear if this is a requirement for all samples. It seems to imply that it is, please verify.**

SIM analysis is a requirement of this solicitation. Laboratories must show evidence during the solicitation process that they are capable of providing SIM analysis for Polyaromatic Hydrocarbon (PAH) compounds in the semivolatile fraction, and for 1,4-dioxane, ethylene dibromide, and dibromo-chloropropane in the volatile fractions. Any other SIM requests than these will be incorporated as modified analyses during the tenure of the contracts and will be handled on a case-by-case basis.

16. **Statement: This solicitation does not appear to have an Inspection of Services and Warranty of Services Clause which was in some of the older CLP contracts. Regions need to have the ability to reject data and reduce payment to laboratories when they perform poorly (e.g., poor PE sample scores which cause data to be rejected).**

This question is related to the RFP and will be addressed by USEPA OAM.

17. **Statement: The methods need to contain definitive contractual language regarding sample dilutions. In the current OLM04.3, the lack of definitive wording has resulted, in some cases, in the Region receiving data of no or little use because of over-dilution of the sample. The methods need to specify that dilutions must keep the responses of the target compounds (which require dilution) in the upper half of the initial calibration range.**

The SOW language throughout the Exhibit Ds been changed from "shall" to "must keep responses of the target compounds (which require dilution) in the upper half of the initial calibration range".

## Exhibit A

1. **Statement: Section 4.2.2.9, p. A-10: According to the analytical sections of the SOW, the NIST (2002 release or later) or equivalent mass spectral library should be used for TIC searches. In addition to the NIST (2002 release or later), this Section includes a reference to the Wiley (1991 release or later) mass spectral library which is not referenced in the analytical portions of the SOW.**

The SOW language has been modified to include the Wiley reference when applicable.

---

## Exhibit B

1. **Statement: 6B - Form VI VOA-2: Xylenes are reported as xylene (total). The SOW requires xylenes to be reported separately as o-xylene and m, p-xylene.**

**6D - Form VI VOA-SIM: Some DMCs are listed incorrectly or are missing: trans-1,3-dichloropropene-d8 should be removed from the form; toluene-d5 should be reported as toluene-d8; and benzene-d6 and 1,2-dichloropropane-d6 should be added to the list.**

**7B - Form VII VOA-2: Xylenes are reported as xylene (total). The SOW requires xylenes to be reported separately as o-xylene and m, p-xylene.**

**7D - Form VII VOA-SIM: Some DMCs are listed incorrectly or are missing: trans-1,3-dichloropropene-d8 should be removed from the form; toluene-d5 should be reported as toluene-d8; and benzene-d6 and 1,2-dichloropropane-d6 should be added to the list.**

The SOW forms have been corrected appropriately.

---

## Trace VOA

1. **Statement: Section 9.4.5.2, p. D-26/Trace VOA, The language in this section and in the footnote to Table 2 suggests that not every TCL and DMC has a required minimum RRF. According to Table 2, each TCL and DMC has a minimum RRF.**

The SOW has been corrected to state that all target compounds and DMCs have a minimum Relative Response Factor (RRF).

2. **Statement: Section 11.1, p. D-31/Trace VOA, there does not appear to be any guidelines for qualitative identification of the target compounds in SIM analysis.**

No specific guidelines for qualitative identification of the target compounds in SIM analysis are required since they will most likely be the same as for the full scan method. If the laboratory finds it necessary to modify the procedure for SIM analysis to meet the guidelines for qualitative identification, it may do so provided that the modifications have been documented in the Sample Delivery Group (SDG) Narrative and all acceptance criteria have been met.

3. **Statement: Section 11.3.4.2, p. D-35/Trace VOA: Dilution does not affect the DMC Percent Recovery since the sample is spiked with the DMC and internal spiking solutions after dilutions are made. The DF (Dilution Factor) should be removed from the equation.**

The SOW has been modified to remove the Dilution Factor (DF) from the equation.

4. **Statement: Section 11.3.5, p. D-36/Trace VOA: To evaluate the internal standards and retention times for samples and blanks analyzed during the same 12 hour time as the initial calibration, this section requires use of a 5 µg/L standard. Please clarify if a 5 µg/L standard or the mid-level calibration standard listed in Section 7.2.2.6.2 (i.e., 5.0 µg/L for non-ketones, 50 µg/L for ketones, 250 µg/L for 1,4-dioxane, 20 µg/L for 1,4-dioxane by SIM technique, and 0.5 µg/L or other compounds analyzed by SIM) is required.**

Clarifying language has been added to Section 11.3.5.

5. **Statement: Section 11.4.4, p. D-36/Trace VOA: The first paragraph of this section contains technical acceptance criteria for the DMCs. The second paragraph of this section contains requirements for the internal standards (EICP areas) and for the DMCs in SIM analysis. It is recommended to include the internal standard requirements in a separate section from the DMC requirements.**

The internal standard requirements have been put in a separate section, Section 11.4.5.

6. **Statement: Table 1, p. D-45/Trace VOA: There seems to be a "typo" in the criteria for mass 175 since it reads "greater that 5.0-9.0%", it should be just "5.0-9.0%".**

The typo has been corrected.

7. **Statement: General comment for SIM Trace VOA analysis: The SOW requires analysis of 14 DMCs for three target compounds: 1,4-dioxane, 1,2-dibromoethane, and 1,2-dibromo-3-chloropropane. It seems that the required number of DMCs is excessive for the number of TCLs specified in the SOW.**

The SOW has been modified to reflect that only the DMCs associated with these three compounds are to be analyzed and recorded. The laboratory may wish to add the complete 14 compound DMC mix to the sample, but only those associated with the three compounds are to be reported.

8. **Statement: General comment for SIM trace VOA analysis: The SOW does not appear to address recommended instrument operating conditions including the selected ions and dwell times, etc. Also, the minimal requirements that exist for SIM analysis are not easily located in the text. The SOW should contain a separate section that is more comprehensive for the SIM analysis.**

SIM analysis does not require a separate Exhibit D for both volatiles and semivolatiles since most of the requirements for SIM are exactly the same or very similar to the full scan methods.

## **Low/Med VOA Method**

1. **Statement: 10.1.6.2, Clarify baseline rise. What is the intent?**

This issue is in reference to the use of dilution in cases where a significant baseline rise or hump-ogram is present.

2. **Statement: Section 6.7.5, p. D-13/Low-Med VOA: This Section specifies that the MS must be capable of meeting the BFB criteria in Table 1 when 20 ng of BFB is injected. According to Section 7.2.2.1, the required concentration of BFB is 50 ng on column.**

Section 6.7.5 has been modified to state that 50 ng of 4-bromofluorobenzene (BFB) is injected.

3. **Statement: Section 7.2.2.6.4, p. D-17/Low-Med VOA: This Section should specify the concentrations of DMC compounds in addition to TCLs.**

Section 7.2.2.6.4 has been modified to specify the concentration for associated DMCs.

4. **Statement: General comment for Section 8.1.1, p. D-18/Low-Med VOA: Containers, numbers of the required/recommended containers, preservation criteria, and directions for the laboratory based on the types of samples received are quite convoluted and difficult to follow. It is recommended to rework this Exhibit as well as include a summary of this information in table format (or a flowchart).**

The SOW has been modified accordingly to be more concise and easier to follow.

5. **Statement: The number of additional containers required for MS/MSD analysis in Section 8.1.1.2 is not sufficient and is inconsistent with previous guidance given by ASB.**

The SOW has been modified to state that eight additional vials are required on low/medium volatile soil samples requiring Matrix Spike and Matrix Spike Duplicate (MS/MSD).

6. **Statement: Sections 9.3.5.2, p. D-26/Low-Med VOA and 9.4.5.2, p. D-29/Low-Med VOA: The language in these sections suggest that not every TCL and DMC has a required minimum RRF. According to Table 4, each TCL and DMC has a minimum RRF.**

The SOW has been modified to state that every target compound and DMC has a required minimum RRF.

7. **Statement: Section 9.4.5.1, p. D-28/Low-Med VOA: This Section incorrectly specifies concentrations of the CCV as 5.0 µg/L for non-ketones, 10 µg/L for ketones, and 250 µg/L for 1,4-dioxane. According to Section 7.2.2.6.4, the concentrations of the TCLs in the CCV are: 50 µg/L for non-ketones, 100 µg/L for ketones, and 1250 µg/L for 1,4-dioxane.**

Section 9.4.5.1. has been modified to state that the concentrations of the target compounds in the Continuing Calibration Verification (CCV) are: 50 µg/L for non-ketones, 100 µg/L for ketones, and 1250 µg/L for 1,4-dioxane.

8. **Statement: Section 9.4.5.3, p. D-28/Low-Med VOA: The reference to Table 2 should be Table 5.**

The reference has been changed to Table 4.

9. **Statement: In Section 10.1.5.6, p. D-35/Low-Med VOA, the SOW includes a step for adding 100 µg/L of methanol extract to the 5mL of reagent water; in Section 10.1.5.7 the requirement for the amount of the reagent water appears to be 4.9 mL and not 5 mL.**

Section 10.1.5.7 has been changed to 5 mL and reflect the addition of 100 ug/L.

10. **Statement: Sections 10.1.6.2 and 10.1.6.4 state that the dilution factor chosen should keep the responses of the volatile target compounds that required dilutions in the upper half of the initial calibration range. This language needs to be changed to must so that all target compounds that require dilutions must be in the upper half of the initial calibration range.**

The language in Section 10.1.6.4 has been changed to from "shall" to "must".

11. **Statement: Section 10.1.6.3 states that when the response of the soil sample analysis exceeds that of the high standard, an appropriate weight less than 5g should be used. This procedure will not work since taking a smaller aliquot will require opening the container, which will release the volatiles. This defeats the purpose of the closed-system volatile method.**

Section 10.1.6.3 of the SOW has been modified to state "...if the response of any target compound in the sample exceeds the response of the same target compound in the high standard, then a new sample must be prepared and analyzed by the medium-level method...".

12. **Statement: The requirements for storage blanks for soil samples varies between sections: Section 8.2.2.1: Aqueous storage blanks shall be stored with preserved low/medium soil samples and water samples within an SDG until all such samples are analyzed. Inert sand storage blanks shall be stored with unpreserved low/medium soil samples until all such samples are analyzed. Could inert sand storage blanks also be used with preserved low/medium samples?**

The SOW has been modified to reflect the following:

"8.2.1 Unpreserved low/medium soil samples must be protected from light and stored at less than -7°C from the time of receipt until time of analysis. Store unused sample aliquots at less than -7°C until 60 days after delivery of a reconciled, complete Sample Data Package to USEPA. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations."

"8.2.2 Sodium bisulfate preserved low/medium soil samples and water samples must be protected from light and stored at 4°C (±2°C), in a refrigerator used only for storage of volatile samples, in an atmosphere demonstrated to be free of all potential contaminants, until 60 days after delivery of a reconciled, complete Sample Data Package to USEPA. After 60 days, the samples may be disposed of in a manner that complies with all applicable regulations."

SOM01.X Comments and Responses  
*Low/Med VOA (Con't)*

"8.2.2.1 Aqueous storage blanks shall be stored [at 4°C (±2°C)] with preserved low/medium soil samples and water samples within an Sample Delivery Group (SDG) until all such samples are analyzed. Inert sand storage blanks shall be stored at less than -7°C with unpreserved low/medium soil samples until all such samples are analyzed."

13. **Statement: Section 12.1.1.2: "Storage Blank - A volume of a clean reference matrix (reagent water for water samples stored at 4°C (±2°C) or inert sand for soil samples stored at less than -7 °C)...". Note: This Section does not address a storage blank for preserved soil samples stored at 4°C (±2°C).**

The following language has been incorporated to address a storage blank for preserved soil samples stored at 4°C (±2°C):

"NOTE: If the SDG contains samples stored at 4°C (±2°C) and samples stored at less than -7°C, two storage blanks will be prepared, one for each condition."

14. **Statement: Section 12.1.2.3: A minimum of one storage blank must be analyzed per matrix type (1 for soil and 1 for water sample) after all samples for the SDG stored in the same manner have been analyzed, unless the SDG contains only ampulated Performance Evaluation (PE) samples. Note: More than one storage blank may be required for soils.**

The following language has been incorporated into Section 12.1.1.2 to address a storage blank for preserved soil samples stored at 4°C (±2°C):

"NOTE: If the SDG contains samples stored at 4°C (±2°C) and samples stored at less than -7°C, two storage blanks will be prepared, one for each condition."

15. **Statement: Section 11.2.4.2, p. D-44/Low-Med VOA, Eq. 13: Dilutions do not affect the DMC Percent Recovery since the sample is spiked with the DMC and internal spiking solutions after dilutions are performed. The DF (Dilution Factor) should be removed from the equation.**

The SOW has been modified to remove DF from the equation.

16. **Statement: Table 1, The note under the Table is incomplete. There seems to be a "typo" in the criteria for mass 175, since it reads "greater than 5.0 - 9.0%". It should be just "5.0 - 9.0%".**

Table 1 has been corrected to indicate "5.0 - 9.0%".

17. **Statement: Table 2 is incomplete. It lists primary quantitation ions for only 29 out of 52 TCLs.**

The primary quantitation ions for the remaining 23 target compounds have been added to Table 2.



18. **Statement: Sec 10.1.6.3, p. D-36/LOW-MED VOA: In order to comply with this section for Encore and pre-preserved samples the %moisture jar would have to be used. Is this acceptable? (Implied in Sec. 10.3, p. D-37/LOW-MED VOA)**

If at all possible, samples should be taken from one of the four Encore™ samplers or pre-preserved samples. The percent moisture jar should only be used as a last resort.

19. **Statement: Section 9.1.1.5, page D-21/LOW-MED VOA allows for the use of a heated purge for aqueous samples. While this would increase production for VOA GC/MS, Section 9.1.1.1, page D-20/LOW-MED VOA requires an unheated purge for medium-level solids. This requirement negates the heated-purge option for aqueous samples since medium-level solids are analyzed using an aqueous initial calibration (Section 9.3.3.4, p. D-24/LOW-MED VOA). Additionally, a heated purge increases the recovery and stability of 1,4-dioxane and late-eluting compounds. Please explain why a heated-purge cannot be used for medium-level solids when it is allowed for low-level solid and aqueous samples.**

The column would most likely become saturated with methanol, potentially overshadowing analyte peaks and broadening analyte peaks.

20. **Statement: The low medium-level volatile method is very confusing, even to those familiar with Method 5035A. Sections describing containers, numbers of required/recommended containers, preservation criteria, and directions for the laboratory based on the types of samples received are quite difficult to follow. For example, the method (section 10.1.6.3) states that when the response of the soil sample analysis exceeds that of the high standard, an appropriate weight less than 5 g should be used. This procedure will not work since taking a smaller aliquot will require opening a container which releases the volatiles. This defeats the purpose of the closed-system volatile method.**

Section 10.1.6.3 of the SOW has been modified to state "...if the response of any target compound in the sample exceeds the response of the same target compound in the high standard, then a new sample must be prepared and analyzed by the medium-level method...".

21. **Statement: When VOAs cannot be preserved due to bubbling, can laboratory holding time be reduced to five days for laboratory since technical holding time of unpreserved samples is seven days? (May not be known ahead of time to deal with by Flex Clause.) Prefer address in SOW. Trace VOA 8.4 Low VOA 8.4.**

There is no technical precedence for five days. If these types of samples are suspected they can be handled through a Modified Analysis request, or submitted as Preliminary Results (PRs).

Section 8.4 has been modified to indicate "Analysis of unpreserved, unfrozen soil/sediment samples must be completed within 24 hours of Validated Time of Sample Receipt (VTSR)."

## SVOA Method

1. **Statement: Confusing/ambiguous, SVOA, D-33: "Note: All soil/sediment samples in a Case must be extracted by same procedure." Does this refer to all samples in a case being extracted as MED level or by Soxhlet sonication? Need to clarify with additional line (i.e., Soxhlet, sonication, etc.).**

Language has been added in Section 10.1.4.4.1 stating that "Note: All medium-level soil/sediment samples in a Case must be extracted by same procedure."

2. **Statement: Section 7.2.4.5.1 states that pentachlorophenol (among others) only require that a four-point calibration since detection less than 10 ng/uL is difficult. With the SIM calibration ranging from 0.2 to 1 ng/uL it is unclear that this compound will be detected with any reliability in the absence of derivatization. Our laboratory has always derivatized this compound at low levels.**

At the present time, USEPA is going to refrain from changing the requirements of the current SOW. However, USEPA will monitor this analyte as requests for analysis are made, and will modify the SOW as appropriate.

3. **Statement: The PE sample mixture used for pre-awards should be at levels such that the laboratories will need to perform SIM analysis, a new analysis for the CLP.**

Performance Evaluation (PE) samples will not be required for SIM analysis.

4. **Statement: The SIM methods lack detail and is not comprehensive. The SOW should contain recommended instrument operating conditions including the selected ions and dwell times, etc. The requirements for qualitative identification should be included since they will differ from the full scan requirements. The SOW is unclear with regard to SIM MS/MSDs since the MS spiking solution and recovery/RPD table list compounds that are neither PAHs or phenols.**

The SOW has been modified to give some recommended instrument operating conditions (i.e., dwell times) but the onus is on the laboratory to adjust the instrument conditions to achieve the necessary technical acceptance criteria. The SOW has been modified for SIM MS/MSD to state that the Matrix Spike spiking solution may contain only acenaphthene, pentachlorophenol, and pyrene.

5. **Statement: The SOW allows Regions the option of requesting MS/MSDs for volatiles and semivolatiles, but not an option to request a laboratory duplicate. Since there are more DMCs than there are matrix spike compounds, the duplicate sample results may provide more information about laboratory precision. The Region may want to request only a laboratory duplicate for certain projects. This would save the cost/effort of one sample analysis. The Region has historically requested this with DMCs.**

For future SOWs or modifications to SOM, USEPA may add language, forms, and calculation instructions to cover laboratory duplicates. Currently, the Region may choose to collect a field duplicate but it will be treated as a separate field sample (i.e., different EPA sample number).

6. **Statement:** The requirement to receive proper approval from the USEPA CLP PO of sample origin to perform more than two dilutions of a sample needs to be changed. Approval should be received from the Region (via the RDCC/SMO channel) as with other issues. The CLP PO of sample origin is not necessarily the laboratory's PO. Note that Section 10.1.6.1 states to contact SMO for further instructions before performing a second dilution. This is the preferred approach. This similar requirement in the inorganic SOW regarding authorization from the CLP PO in requiring pH adjustment is not working well. SOM01.X has been modified to indicate that flexibility is allowed, however a proper MDL study should be performed and provided.

All Exhibit Ds have been modified to state that the laboratories will contact the Region via SMO for guidance on multiple dilutions.

7. **Statement:** Section 9.1.2, p. D-20/SVOA does not include the ionization mode in the Mass Spectrometer conditions.

The ionization mode has been added to indicate Electron Ionization (EI) mode in Section 9.1.2.

8. **Statement:** Section 9.4.5.5, p. D-26/SVOA: "For the optional analysis of PAHs/phenols using the SIM technique, up to two semivolatile target compounds may fail to meet the criteria listed in Sections 9.4.5.2 and 9.4.5.3 for the opening CCV". This is different from the initial calibration criteria in Section 9.3.5.5 which states: "For the optional analysis of PAHs/phenols using the SIM technique, one PAH compound and/or one phenolic compound may fail to meet the criteria in Sections 9.3.5.2 and 9.3.5.3.

Up to two compounds can fail criteria in Section 9.3.5.2 and up to two compounds can fail criteria in Section 9.3.5.3.

9. **Statement:** Section 10.1.4.5.4, p. D-33/SVOA, specifies to "add 0.4 µg/L of each SIM DMC" to the medium-level soil samples. Why would a SIM analysis be performed on a medium-level soil sample?

The SOW has been modified to reflect that SIM DMCs are not required for medium-level soil samples.

10. **Statement:** Section 11.2.4.2, p. D-48/SVOA, Eq. 9: To calculate the DMC Percent Recovery, the DF (Dilution Factor) should be in the numerator and not the denominator.

The SOW has been modified to place the DF in the numerator.

11. **Statement:** Section 12.2.3.3, p. D-55/SVOA: specifies to add DMCs and matrix spike compounds to the medium-level soil MS and MSD samples to result in the addition of 400 µg of each DMC and MS compound. The amount added should result in the addition of 40 ug not 400 ug.

Section 12.2.3.3 has been modified to state "add DMCs and matrix spike compounds to the medium-level soil MS and MSD samples to result in the addition of 40 µg of each DMC and MS compound."

12. **Statement:** Section 12.2.3.3 p. D-55/SVOA, says to proceed with the extraction procedure in Section 10.1.4.5, which specifies to add 9.5 mL of methylene chloride. This volume should be reduced for the medium-level MS and MSD samples to account for the volume added from the addition of the MS spiking solution.

The SOW has been modified to account for the volume added for the addition of the Matrix Spike spiking solution.

13. **Statement:** Table 1, p. D-59/SVOA, lists the ion abundance criteria for DFTPP. The criteria for masses 68 and 70 are based on the abundance of mass 69, yet mass 69 is not listed in the table. Mass 69 should listed with an ion abundance criteria of "present". The ion abundance criteria for mass 442 is listed as "greater than 50.0% but less than 100% of mass 198". This is inconsistent with the note which states that mass 442 "may be up to 100% that of m/z 198". To be consistent, the table should list the ion abundance criteria of mass 442 as greater than 50.0% but less than or equal to 100% that of m/z 198.

The SOW has been modified to correct the ion abundances.

14. **Statement:** Section 12.1.5.5: "If the method does not meet the Retention Time (RT) requirements for... the DMCs (Section 11.3.5)... Sample analysis cannot proceed until the method blank meets these requirements." There does not appear to be RT criteria for DMCs and Section 11.3.5 refers to IS area responses.

The SOW has been modified to remove language regarding Retention Time (RT) criteria for DMCs.

15. **Related Statement:** The SOW does not adequately address the SIM method. The SOW should contain recommended instrument operating conditions including the selected ions and dwell times, etc. The requirements for qualitative identification should be included since they will differ from the full scan requirements. The SOW is unclear with regard to SIM MS/MSDs since the MS spiking solution and recovery/RPD table list compounds that are not PAHs or phenols. The SOW should also include a statement that no qualitative identification of non-target compounds is necessary for SIM analysis. Perhaps the SOW should contain a separate Exhibit D for the optional SIM analysis of PAHs and phenols.

The laboratory will have the option of using the same Matrix Spike solution or one containing only the PAHs. There is no need to break out SIM analyses in a separate Exhibit D.

16. **Statement:** What is the correct concentration of Internal Standard Solution in sample extracts? In Exhibit D: Analytical Methods, the concentration is listed as 40ng/uL in sections 7.2.4.6.1, 10.6.2, 10.6.4, and 10.6.5, and 20 ng/uL in sections 9.3.3.3, 9.4.3.2, and 10.6.6.1. We now use 20 ng/uL for all scanning MS methods, including OLM04.3, OLC03.2, and 8270. We can perform an analysis with either value, but not with both at the same time.

The SOW has been corrected to indicate that the concentration for Internal Standards is 20 ng/uL.

17. **Statement: Is it known that the new target compound 2,3,4,6-Tetrachlorophenol has an incorrectly listed CAS number? The correct CAS number is 58-90-2 (from Exhibit C, Target Compound List).**

The SOW has been corrected to indicate that CAS Number for 2,3,4,6-Tetrachlorophenol is 58-90-2.

18. **Statement: What type of instrumentation and columns have proven to deliver consistent reporting limits of 0.2 ppb for PCB on typical CLP samples? Can you please explain the amount of testing done to support this requirement? Has consideration been given to increase the level to 2 ppb, which CEIMIC believes may be achievable?**

The instrumentation and the columns listed in Exhibit D, Aroclors have been shown by many laboratories to deliver consistent reporting limits of 1 ppb or lower. This is the same CRQL as in the OLM04.2 SOW.

---

## Pesticides Method

1. **Statement: Section 7.2.3.3, If the resolution check mixture is not commercially available may we substitute the concentration of TCX. If we compare the ratios of the analytes in the calibration standards we will note the TCX and DCB are 1:2, while in the "RES CHECK" they are 1:1. This reduces cost based on the standards and resolution check being able to come from the same intermediate stock. Can't we just exclude the resolution check since our group C standards will already be at those concentrations? This will also be a cost-reducing factor. Also, we don't have to run the initial because our standards already contain everything at the necessary concentrations.**

For convenience in purchasing standards, the concentration of tetrachloro-m-xylene (TCX) in the Resolution Check Mixture will be lowered from 20 ng/mL to 10 ng/mL. The requirements for sequences and the use of specific calibration standards will not change from the current SOW.

2. **Statement: Must we use a final volume of 10 mL with a standard? Current technologies would allow for a lower standard and perhaps less concentrated samples. Reduction of standard wastes and less pollution via blow down and such.**

The volume of the standard does not really matter as long as the appropriate analyte concentrations are being analyzed. The 10 mL volume for standards is meant as a guideline.

3. **Statement: Why can't we use acetone/hexane for extractions? With the concerns of environmental problems I would see this as an option. Our Institute SWRI Environmental Management System (EMS) states, "Continually improve environmental performance and prevent pollution through process efficiency and improved technologies." DCM is considered a group B2 carcinogenic while both acetone and hexane are not, by USEPA terminology. The use of volumetric flasks is still allowed in SOM01.X. However, appropriate language has been incorporated into SOM01.X to indicate that vacuum tubes are also permitted. The volumetric flasks must have a Class A rating.**

As the CLP attempts to stay consistent with methods published in SW-846, USEPA will not be able to require alternate solvents for extraction. As different solvents are evaluated and the effectiveness of each is validated, CLP will try to incorporate their use.

4. **Statement: Why not split the sample for pesticides and PCBs, if both analyses are needed, and do an acid clean up on the extract for PCBs? We would have to do separate spikes for the LCSs and MS/MSD.**

The current SOW does not prohibit the splitting of the pesticide and Aroclor fractions if both analyses are requested, as long as the required CRQLs can be achieved and appropriate cleanup actions are performed.

5. **Statement: Section 6.26.1.3 states, "One wide-bore fused silica GC column may be used provided that the resolution requirements are met (see Section 9.2.5.2)". Section 9.2.5.2 specifies resolution criteria for both primary and confirmation columns. Section 10.4.1.1 states "Sample analysis on two different non-equivalent GC columns is required for all samples and blanks (See Section 6.26.1.3)". Two GC columns are specified throughout the SOW so that statement in Section 6.26.1.3 may be problematic.**

Section 6.26.1.3 of the SOW has been modified to reflect, "Wide-bore (0.53mm ID) fused silica GC column may be used provided that the resolution requirements are met (see Section 9.2.5.2).".

6. **Statement: Section 9.2.4.2 specifies to calculate the mean absolute RT for each single component pesticide and surrogate but does not specify to do so for the Toxaphene peaks. Section 9.2.4.3, however, specifies that the RT windows for Toxaphene are centered around the mean absolute RT, which was never calculated.**

Section 9.2.4.2 has been modified to include language regarding Toxaphene peaks.

7. **Statement: Section 10.3.2.2.3, p. D-50/PEST, Eq. 13: To calculate the Percent Recovery, the DF (Dilution Factor) should be in the numerator and not the denominator.**

The SOW has been modified to place DF in the numerator.

8. **Statement: Section 11.4.3 refers to "...surrogate recoveries outside the lower advisory surrogate acceptance limits...". The surrogate recoveries are not advisory.**

Section 11.4.3 has been modified to reflect "...surrogate recoveries outside the lower surrogate acceptance limits...".

9. **Statement: The new contract requires different concentrations for the surrogate spiking solution. It is more concentrated and also a larger volume is being spiked into the samples. It is barely okay for water samples, but for soil samples the surrogate concentration in the extract will exceed the linearity range of the ICAL (0.12 ppm TCX and 0.24 ppm DCB in the extract). I doubt they are aware of this problem and I think someone should bring it to their attention.**

The required surrogate concentrations in the extract have been modified to be 12 times the concentration of the low standard [0.060 ppm TCX and 0.12 ppm Decachlorobiphenyl (DCB) in the extract] for both pesticides and Aroclors.

10. **Statement: Discrepancy in Pesticides-Section 10.3.1.4.5, mentions GPC Pattern for Toxaphene, but Toxaphene is not included in GPC Standards 7.2.3.8.1.**

The SOW has been modified to remove language regarding Toxaphene.

11. **Statement: Florisil studies on clean-up for pesticides-when was this performed? How much difference does it make to use Florisil clean-up? Our studies show little impact on extracts.**

CLP has historically used Florisil cleanup for pesticides, and will continue to require this for this SOW. No current studies have been performed, but the consensus of experts within USEPA believe it is useful in removing potential interferences in the pesticide fractions.

---

## Aroclors Method

1. **Statement: Aroclor 1268 contains DCB. What about the calibration standards? QL-DCB will be up when analyzing Aroclor 1268.**

Because the SOW requires the laboratory to perform an initial calibration on Aroclors 1016 and 1260, unless other Aroclors are detected in field samples, this will only be an issue when the laboratory has to perform an initial calibration for Aroclor 1268. If the laboratory has to calibrate for Aroclor 1268, and the Percent Recovery for the DCB is shown to be high, then the laboratory can note the fact there is contribution of DCB from the Aroclor 1268 in the Case narrative. The laboratory can also use the recovery results of TCX to validate the analysis (the SOW gives an allowance of one of the surrogates failing).

2. **Statement: 9.3.4.3: The opening CCV, the CF for each Aroclor peak and surrogates calculated from the CCV standard must not exceed a difference of more than  $\pm 15\%$  when compared to the CFs from the initial calibration curve. For the closing CCV, the CFs for each Aroclor peak and surrogates calculated from the CCV, standard must not exceed a difference of more than  $\pm 50\%$  when compared to the CFs from the initial calibration curve. If the difference for the closing CCV is  $\pm 15\%$ , then it can be used for the opening CCV of the next 12-hour. This is strange.**

If USEPA were to make the closing CCV criteria the same as the opening CCV criteria this could put undue hardship on the laboratory. Therefore, USEPA is relaxing the requirements of the closing CCV in an attempt for higher level Quality Assurance (QA) without putting too much burden on the laboratory. As USEPA reviews the results, the closing CCV requirement may be made more stringent, as appropriate.

3. **Statement: Is the recovery of Aroclor 1016 upper limit of 120% correct? See Table 2.**

The SOW has been modified to indicate an upper limit of 150%.

SOM01.X Comments and Responses  
*ARO (Con't)*

4. **Statement: There is no nitrogen evaporation device listed in Section 6, Equipment and Supplies, yet Section 10.1.5.3.2 describes concentration using a nitrogen evaporation technique.**

The SOW has been modified to include a nitrogen evaporation device in Section 6.8.

5. **Statement: Section 9.3.1 under the Summary of Continuing Calibration Verification (CCV) states "...data are not acceptable unless bracketed by acceptable analyses of instrument blanks, PEMs, and the Aroclor CS3 Standard Mixtures". The PEMs should be removed from this section.**

The SOW has been modified to remove the Performance Evaluation Mixtures (PEMs) from this section.

6. **Statement: The end of Section 9.3.2.6 includes the statement "...whichever was due to be performed at the end of the 12-hour period." This part of the sentence should be deleted since it does not apply to the Aroclor analysis sequence.**

Section 9.3.2.6 of the SOW has been modified to remove this part of the sentence.

7. **Statement: Section 11.2.4.1, p. D-51/ARO, Eq. 13: To calculate the DMC Percent Recovery, the DF (Dilution Factor) should be in the numerator and not the denominator.**

Section 11.2.4.1 of the SOW has been modified to place the DF in the numerator.

8. **Statement: Section 11.3.6 states that up to one surrogate may fail this criteria. This is different from the Pesticide SOW which states that up to one surrogate may fail criteria, per column.**

Section 11.3.6 has been modified to indicate that up to one surrogate may fail this criteria, per column.

9. **Statement: Section 11.4.3 refers to "...surrogate recoveries outside the lower advisory surrogate acceptance limits...". The surrogate recoveries are not advisory.**

Section 11.4.3 has been modified to indicate "...surrogate recoveries outside the lower surrogate acceptance limits...".

10. **Statement: Section 12.3.3.1, Water LCS, specifies to "...measure two additional 1L aliquots...". Only one aliquot is necessary for the LCS.**

Section 12.3.3.1 has been modified to indicate "...measure one 1 L of reagent water...".

11. **Statement: Section 12.3.3.2, Soil/Sediment LCS, specifies to "...measure two additional 30g...". Only one aliquot is necessary for the LCS.**

Section 12.3.3.2 has been modified to indicate "...measure out 30 g of a clean reference matrix...".



12. **Statement: Table 1, MS/MSD Recovery and RPD Limits, does not specify the matrix. Table 1 should state that the limits are for both soil and water, as Table 2 does. Also, how were these limits derived?**

Table 1 has been modified to specify limits for both soil and water samples. These limits may eventually be adjusted if the limits are deemed to be inappropriate.

13. **Statement: Section 11.3, Sample Technical Acceptance Criteria, does not include the specification that the LCS associated with the samples must meet the LCS technical acceptance criteria. Section 12.3.6.3, however, has specifications regarding the treatment of samples associated with LCSs that do not meet LCS acceptance criteria. This requirement should also be placed in Section 11.3.**

The SOW has been modified to include this requirement in Section 11.3.3.

14. **Statement: Equation 2, p. D-21/ARO, uses peak area or height in the calculation of the CF. Equation 6, p. D-47/ARO, defines Ax as the Area (not or height). This could be problematic if height was used in Eq. 2 but area was used in Eq. 6. Also, Section 9.2.4.4, p. D-20/ARO, states that either peak area or peak height may be used to calculate Cfs.**

Equation 6 has been modified to define Ax as "Area or height of the peak for the compound to be measured."

15. **Statement: The note for Vo in Equation 6, p. D-47/ARO, refers to "instrument sulfur blanks". It should refer to "instrument and sulfur cleanup blanks".**

Equation 6 has been modified to indicate that  $V_o$  refers to "instrument and sulfur cleanup blanks".

16. **Statement: Section 9.2.1 under Summary of Initial Calibration states, "The other seven Aroclors are calibrated at a single mid-point for pattern recognition. The standards for these five Aroclors should be analyzed..." The second part should state "The standards for these seven Aroclors should be analyzed..."**

Section 9.2.1 has been modified to indicate "The standards for these **seven** Aroclors should be analyzed..."

17. **Statement: Section 7.2.4.4.1 and the Table 3 footnote specify TCX and DCB concentrations that conflict with the values in Table 3. Section 7.2.4.4.1 lists the concentrations for TCX and DCB in the calibration standards as 5, 10, 20, 40, 80 µg/L and 10, 20, 40, 80, 160 µg/L, respectively, while Table 3 lists the concentrations as 100, 200, 400, 800 and 1600 µg/L for both TCX and DCB. The note for Table 3 lists the TCX and DCB concentrations in the CS3 standard as 20 and 40 µg/L, respectively, while the tables lists them both as 400 µg/L.**

The table has been corrected to list the concentrations for TCX and DCB in the calibration standards as 5, 10, 20, 40, 80 ng/mL and 10, 20, 40, 80, 160 ng/mL.

SOM01.X Comments and Responses

*ARO (Con't)*

18. **Statement: Discrepancy in Aroclors-Section 9.3.1, compared to Section 7.2.3.4.2. - Mid Point Standard vs. low-level standard, for Aroclors 1221, 1232, 1242, 1248, 1254, 1262, and 1268.**

The SOW has been corrected to indicate Mid-Point Standard.

19. **Statement: Due to possible interferences in the Aroclor may more than five peaks be used?**

If a laboratory can adequately modify the proper forms, and adequately provide for the electronic deliverables, then it would be acceptable to report more than five peaks. All peaks reported will be evaluated by Contract Compliance Screening (CCS) (i.e., not just the best 5).